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IS 354-4 (1986): Methods of Sampling and Test for Resins for Paints, Part 4: Special Test Methods for Epoxy Resins [CHD 20: Paints, Varnishes and Related Products]



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“Knowledge is such a treasure which cannot be stolen”

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IS : 354 (Part 4) - 1986

Reaffirmed - 2012

Indian Standard

**METHODS OF
SAMPLING AND TEST FOR
RESINS FOR PAINTS**

PART 4 SPECIAL TEST METHODS FOR EPOXY RESINS

(*Second Revision*)

Second Reprint OCTOBER 2000

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

AMENDMENT NO. 2 JUNE 2003
TO
IS 354 (PART 4) : 1986 METHODS OF SAMPLING AND
TEST FOR RESINS FOR PAINTS

PART 4 SPECIAL TEST METHODS FOR EPOXY RESINS

(Second Revision)

(*Page 5, clause 5*) — Substitute the following title for the existing:

'5 DETERMINATION OF EPOXIDE CONTENT

(*Page 6, clause 5.1.3*) — Substitute the following for the existing clause:

'5.1.3 Calculation

$$\frac{\text{Epoxide content, g equivalent of } (V_1 - V_2) N}{\text{epoxy groups/100 g of resin}} = \frac{10 \times M}{10 \times M}$$

where

V_1 = volume, in ml, of hydrogen bromide used in determination with the material;

V_2 = volume, in ml, of hydrogen bromide used in blank;

N = normality of hydrogen bromide solution; and

M = mass in, g, of the material taken for test.'

(CHD 20)

AMENDMENT NO. 1 JANUARY 1990
TO
IS : 354 (Part 4) - 1986 METHODS OF SAMPLING
AND TEST FOR RESINS FOR PAINTS
PART 4 SPECIAL TEST METHODS FOR EPOXY RESINS
(*Second Revision*)

(*Page 3, clause 0.2, line 7*) — Delete the words 'chlorinated rubber'.

(*Page 3, clause 0.2, last line*) — Delete 'Part 8'.

(CDC 50)

Indian Standard
METHODS OF
SAMPLING AND TEST FOR
RESINS FOR PAINTS

PART 4 SPECIAL TEST METHODS FOR EPOXY RESINS

(Second Revision)

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IS : 354 (Part 4) - 1986

(Continued from page 1)

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Indian Standard

METHODS OF SAMPLING AND TEST FOR RESINS FOR PAINTS

PART 4 SPECIAL TEST METHODS FOR EPOXY RESINS

(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Part 4) (Second Revision) was adopted by the Indian Standards Institution on 18 August 1986, after the draft finalised by the Raw Materials for Paint Industry Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was originally published in 1952 covering methods of sampling and general test methods mainly for natural resins. Subsequently, an Indian Standard for methods of sampling and test for natural and synthetic resins was published as Part 2 of the above standard in 1971. These two parts were amalgamated and revised in 1976. This revision has been necessitated as more and more newer synthetic resins like polyamides, polyvinyls, chlorinated rubber and emulsion polymers are being manufactured and used in the country. While revising the standard, the Committee felt it appropriate to publish this standard in various parts, as indicated below:

- Part 1 General test methods
- Part 2 Special test methods for alkyd resins
- Part 3 Special test methods for phenolic resins
- Part 4 Special test methods for epoxy resins
- Part 5 Special test methods for polyamide resins
- Part 6 Special test methods for amino resins
- Part 7 Special test methods for determination of monomer content in acrylic or vinylacetate containing polymers and emulsions
- Part 8 Special test methods for chlorinated rubber

IS : 354 (Part 4) - 1986

0.3 In this standard (Part 4), test methods covered in **20.1** to **20.3** of IS : 354-1-976* have been included. In addition to above, alternative method for determination to epoxy resins, total chlorine content and hydroxyl value have been added.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

1. SCOPE

1.1 This standard (Part 4) prescribes the special test methods for epoxy resins used in paints and enamels.

2. TERMINOLOGY

2.1 For the purpose of this standard (Part 4), the definitions given in IS : 1303-1983‡ and IS : 6667-1972§ shall apply.

3. SAMPLING

3.1. Representative samples of the epoxy resins shall be drawn as prescribed in 3 of Part 1 of this standard.

4. IDENTIFICATION

4.1 Dissolve one gram of the material without heating in 100 ml of 95 percent sulphuric acid and carry out the following tests.

4.1.1 Add 1 ml of concentrated nitric acid to 1 ml of the solution obtained in **4.1** and shake. Allow to stand for 5 minutes. Pour the mixture into 100 ml of 1 N sodium hydroxide solution. Development of an orange red colouration confirms the presence of epoxy resin.

4.1.2 Add 3 drops of 40 percent formaldehyde solution to the solution of the material in a test tube. Development of a red brown colour which on dilution with water turns green, confirms the presence of epoxy resin.

4.1.3 Prepare Denige's reagent by dissolving 5 g of yellow mercuric oxide in a mixture of 20 ml concentrated sulphuric acid and 100 ml of water and filter. Add 5 ml of the reagent thus prepared to the solution of the material in a test tube. Appearance of an orange precipitate confirms epoxy resin.

*Methods of sampling and test for resins for paints (*first revision*).

†Rules for rounding off numerical values (*revised*).

‡Glossary of terms relating to paints (*second revision*).

§Glossary of terms used in synthetic resin industry.

4.1.4 Add 5 ml of 10 percent (*m/v*) solution of ferric chloride to the solution of the material. Appearance of a dark violet colouration confirms epoxy resin.

5. DETERMINATION OF EPOXY CONTENT

5.0 Outline of the Methods — Two methods are prescribed for determination of epoxy content. In the first method, the material is dissolved in a suitable solvent and titrated against a standard solution of hydrogen bromide in glacial acetic acid. The second method which may be used as alternate method is based on the reaction with hydrochloric acid in pyridine which opens up epoxy ring to provide chloropyrine. Unreacted hydrochloric acid is estimated by back titration.

5.1 First Method

5.1.1 Reagents

5.1.1.1 Chlorobenzene

5.1.1.2 Chloroform-chlorobenzene mixture — 1 : 1 (*v/v*).

5.1.1.3 Crystal violet indicator solution — 0.1 percent (*m/v*) solution of crystal violet in glacial acetic acid.

5.1.1.4 Glacial acetic acid — conforming to IS : 695-1986*.

5.1.1.5 Standard hydrogen bromide in acetic acid solution — 0.1 N. Prepare by bubbling anhydrous hydrogen bromide at a slow rate through glacial acetic acid until the desired normality is attained (approximately 8 g of hydrogen bromide per litre). Standardize the solution against standard potassium phthalate solution prepared by dissolving 0.4 g of the material in 10 ml of glacial acetic acid. Calculate the normality of hydrogen bromide in acetic acid as follows:

$$\frac{M \times 1\,000}{204.2 \times V}$$

where

M = mass in g of potassium phthalate used, and

V = volume in ml of hydrogen bromide in acetic acid used.

5.1.2 Procedure — Weigh the desired amount of the material into a long-necked flask. Dissolve the sample in a solvent at room temperature. Use 10 ml of chlorobenzene for liquid resins and 25 ml of 1:1 mixture of chloroform and chlorobenzene for solid resins. Use may be made of magnetic or suitable stirrers to effect solution. Add 4 to 6 drops of the

*Specification for acetic acid (*third revision*).

IS : 354 (Part 4) - 1986

indicator solution and attach the flask to the rubber stopper on the burette tip. Lower the burette tip just above the solution and titrate against standard hydrogen bromide in glacial acetic acid to a blue green end point, with stirrer rotating at a moderate speed to avoid splashing. Slow down the titration at the end point to allow time for reaction. Conduct a blank determination simultaneously using all the reagents except the sample.

5.1.3 Calculation

$$\text{Epoxy content, percent by mass} = \frac{(V_1 - V_2) N}{10 \times M}$$

where

V_1 = volume in ml of hydrogen bromide used in the determination with the material,

V_2 = volume in ml of hydrogen bromide used in blank,

N = normality of hydrogen bromide solution, and

M = mass in g of the material taken for test.

5.2 Second Method

5.2.1 Reagents

5.2.1.1 Hydrochloric acid — 0.1 N. Dissolve 16 ml of concentrated hydrochloric acid in 1 litre of pyridine in a 1000 ml flask.

5.2.1.2 Potassium hydroxide in alcohol — 0.1 N.

5.2.1.3 Ethyl alcohol — See IS : 323-1959*.

5.2.1.4 Bromothymol blue indicator — 0.1 percent (see IS : 2263-1979†).

5.2.2 Procedure — Weigh 0.5 g in case of liquid epoxy and 1 g in case of solid epoxy resin in a flat-bottom flask. Pipette 25 ml of pyridine hydrochloric acid mixture in the flat-bottom flask and fit with air condenser. Reflux over steam bath for half-an-hour with occasional stirring. Remove the flask with air condenser at the end of this period and cool it down to room temperature. Rinse down the condenser with at least 20 ml of ethyl alcohol and remove the air condenser. Titrate against standard alcoholic potassium hydroxide solution using bromothymol blue indicator near the end point. The change of colour is yellow green to blue. A blank is also carried out without the sample simultaneously as indicated above.

*Specification for rectified spirit (revised).

†Methods of preparation of indicator solutions (first revision).

5.2.3 Calculation

$$\text{Epoxy content, percent by mass} = \frac{(V_1 - V_2) N}{10 \times M}$$

where

V_1 = volume in ml of standard potassium hydroxide solution used in the determination of blank,

V_2 = volume in ml of standard potassium hydroxide solution used in the determination of sample,

N = normality of standard potassium hydroxide solution, and

M = mass in g of the material taken for test.

6. DETERMINATION OF HYDROLYZABLE CHLORINE CONTENT

6.0 Outline of the Method — The material is refluxed in the presence of a known amount of 0.1 N alcoholic potassium hydroxide solution. The amount of potassium hydroxide consumed in the hydrolysis is a measure of the hydrolyzable chlorine content.

6.1 Apparatus**6.1.1 Reflux Apparatus****6.2 Reagents**

6.2.1 Standard Hydrochloric Acid — 0.1 N.

6.2.2 Phenolphthalein Indicator Solution — 0.1 percent (see IS : 2263-1979*).

6.2.3 Alcoholic Potassium Hydroxide Solution — Dissolve 5.5 to 6.0 g of potassium hydroxide in 1000 ml of ethyl alcohol.

6.2.4 Toluene — conforming to IS : 1839-1961†.

6.3 Procedure — Weigh 6 to 8 g of the material accurately and transfer to a 250-ml long-necked flask. Pipette 50 ml of alcoholic potassium hydroxide solution into the flask and add 15 ml of toluene. Stopper the flask, swirl to mix the contents. Attach the reflux condenser and reflux gently for 15 ± 1 minutes. At the end of this period, remove the flask, cool to room temperature. Rinse down the condenser, remove the condenser and add a few drops of the phenolphthalein indicator. Titrate the solution against standard hydrochloric acid to an end point when the colour of the solution changes from pink to colourless.

NOTE - About 100 ml of methyl ethyl ketone may be added to the material, if required, to ensure a homogeneous solution during titration of excess potassium hydroxide solution.

*Methods of preparation of indicator solutions (*first revision*).

†Specification for toluene, reagent grade.

6.4 Calculation

$$\text{Hydrolyzable chlorine (as Cl) ,} \\ \text{percent by mass} = \frac{(V_2 - V_1) N \times 3.55}{M}$$

where

V_2 = volume in ml of standard hydrochloric acid required for titration of the material,

V_1 = volume in ml of standard hydrochloric acid required for titration of the blank,

N = normality of standard hydrochloric acid, and

M = mass in g of the material taken for test.

7. DETERMINATION OF TOTAL CHLORINE CONTENT

7.0 Outline of the Method — The resin is oxidized with sodium peroxide in Method A and with gaseous oxygen in Method B followed by electrometric or volumetric titration of the resulting chlorides.

7.1 Method A — Combustion in a bomb.

7.1.1 Reagents

7.1.1.1 Standard silver nitrate solution — 0.1 N.

NOTE — For testing resins of low total chlorine content [less than 2 percent (m/m)], a standard silver nitrate solution of normality 0.05 N may be used.

7.1.1.2 Dilute nitric acid — 1:9 (v/v).

7.1.1.3 Nitric acid — concentrated (*see* IS : 264-1976*).

7.1.1.4 Sodium peroxide — granulated.

7.1.1.5 Starch or sucrose — as combustion aids.

7.1.2 Apparatus

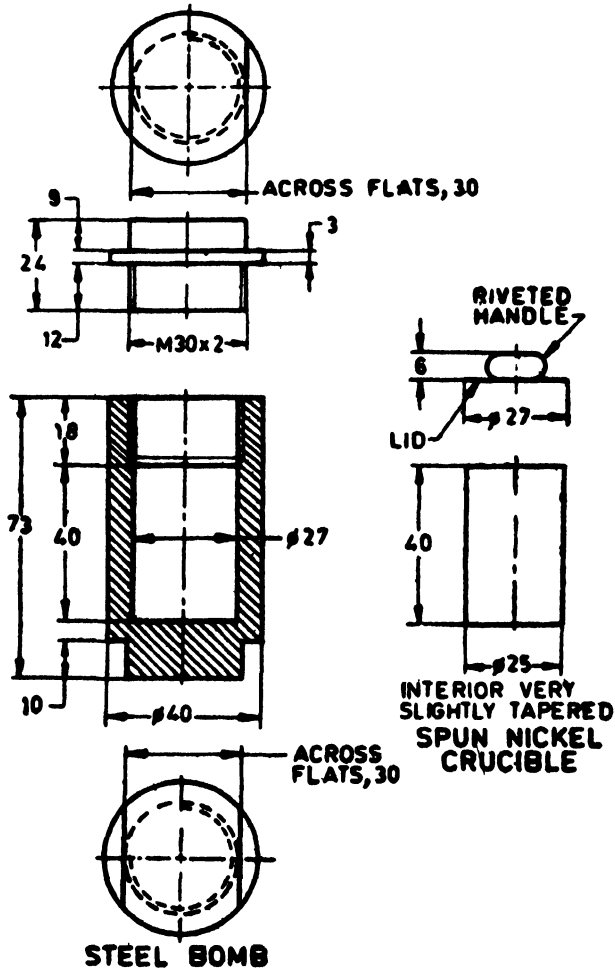
7.1.2.1 Balance — to weigh to an accuracy of 0.000 l. g.

7.1.2.2 Equipment for Volhard or electrometric titration — with a burette.

7.1.2.3 Combustion bomb — (for example, Parr bomb or another bomb which gives the same results), electrically-fired or gas-fired. A suitable gas-fired bomb is shown in Fig. 1.

7.1.2.4 Nickel crucible — with lid, to fit into the bomb (gas-fired). Suitable dimensions are: diameter 25 mm, height 40 mm.

*Specification for nitric acid (*second revision*).



All dimensions in millimetres.

FIG 1 COMBUSTION BOMB, GAS-FIRED TYPE

7.1.2.5 Safety even

7.1.2.6 Beaker — capacity 600 ml.

7.1.3 Test Sample

7.1.3.1 If the sample is in the solid state, it shall be in powdered or granular form, or if necessary shall be cut into pieces 1 to 3 mm in size. When an electrically fired bomb is used, it is preferable that the material be powdered.

IS : 354 (Part 4) - 1986

7.1.4 Procedure — Weigh, to the nearest 0.000 5 g, 0.1 g maximum of the test sample. First place 7 to 7.5 g of the sodium peroxide in the crucible (for the gas-fired bomb), or in the fusion cup of the bomb (for the electrically-fired bomb), then add the test portion mixed with 0.16 to 0.17 g of the starch or sucrose, then an additional 7 to 7.5 g of the sodium peroxide. The filling with sodium peroxide should be carried out behind a shield protecting the operator. Mix all components by stirring, then place the crucible, with the lid in position, inside the bomb and close the bomb tightly. If an electrically-fired bomb is used, assemble the bomb and tap it to settle the charge. Fire the bomb. Cool the bomb. Open it and, if a gas-fired bomb is used, remove the crucible carefully place it in 100 ml of water in the beaker and immediately cover the beaker with a watch glass. When the reaction has subsided, wash down the inside of the bomb and the plug, collecting the washings in the beaker. If an electrically-fired bomb is used, dismantle it after cooling, remove the head and tip it into 100 ml of water in the beaker. Lay the fusion cup in the same beaker and immediately cover with a watch glass. Heat the beaker and its contents to boiling, then cool. Rinse the crucible and lid, or the fusion cup and head, into the beaker with water, then remove them. Slowly add 20 ml of the concentrated nitric acid stirring constantly, followed by the nitric acid solution until the mixture is neutral using methyl orange as indicator. Then add an additional 2 ml of the nitric acid solution. Dilute the contents of the beaker to about 200 ml with water and determine the chlorine content, by electrometric titration or by the Volhard method, with the silver nitrate solution.

7.1.4.1 Blank test — Carry out a blank test by firing the same amount of the sodium peroxide and starch or sucrose as was used with the test portion, and repeating the procedure but omitting the test portion specified in 7.1.3.

IMPORTANT WARNING — 1) If a gas-fired bomb is used, place the bomb in the safety oven. Adjust the flame beforehand, using an empty bomb in the safety oven, so that the top of the flame is a few millimetres from the base of the bomb. Then remove the empty bomb. Heat the test bomb to 300 to 400°C for about 10 min. Ignition usually starts at 50 to 60°C, and is detected by a cracking sound, and the fact that the bottom of the bomb starts to glow.

2) The heating of the bomb is of extreme importance because underheating will cause incomplete oxidation of the organic matter and low chlorine results, whereas overheating may cause damage to the bottom cup and even an explosion.

3) These bombs are not intended to operate at a red heat, but there is no serious danger involved in this method of oxidation provided that the proper charges are used in a bomb that is in satisfactory condition, and that the proper heating technique is used.

4) Discard the bomb if the sides or bottom become visibly swollen, or if the interior surfaces become worn or corroded at any point to an inside diameter 1.5 percent below the original value.

5) It is essential to read carefully the manufacturer's instructions, particularly with reference to safety precautions.

6) When doubt exists as to whether the reaction has taken place, do not dissolve the contents of the bomb into water according to the normal procedure because this might lead to a violent explosion. The contents of the bomb should be spread out onto dry sand, after which they should be sprayed with water from a safe distance and then washed with more water.

CAUTION — If the bomb is cooled in water, take care that the water does not reach the joint between the plug and the bomb.

7.2 Method B — Combustion in a flask.

7.2.1 Reagents

7.2.1.1 Standard silver nitrate solution — 0.1 N.

NOTE — For testing resins of low total chlorine content [less than 2 percent (m/m)], a standard silver nitrate solution of normality 0.05 N may be used.

7.2.1.2 Dilute nitric acid — 1:9 (v/v).

7.2.1.3 Oxygen

7.2.1.4 Sodium nitrate

7.2.1.5 Potassium hydroxide solution — 100 g/l.

7.2.1.6 Hydrogen peroxide solution — 300 g/l.

7.2.2 apparatus

7.2.2.1 Balance — to weigh to an accuracy of 0.000 1 g

7.2.2.2 Equipment for Volhard or electrometric titration — with a burette.

7.2.2.3 Round-bottomed flask — capacity 500 ml, with head for oxygen combustion (see Fig. 2). A platinum wire 1 mm in diameter and 120 mm long in the shape of a tapered spiral is attached to the stopper, a suitable spiral being 15 mm in diameter and 15 mm long. It is recommended that metal gauze be wrapped around the flask for safety or that a safety cage be used.

NOTE — A safety bulb can be fitted to the flask to permit expansion of the gases to reduce the hazard.

7.2.2.4 Filter paper — free from halogens and ash.

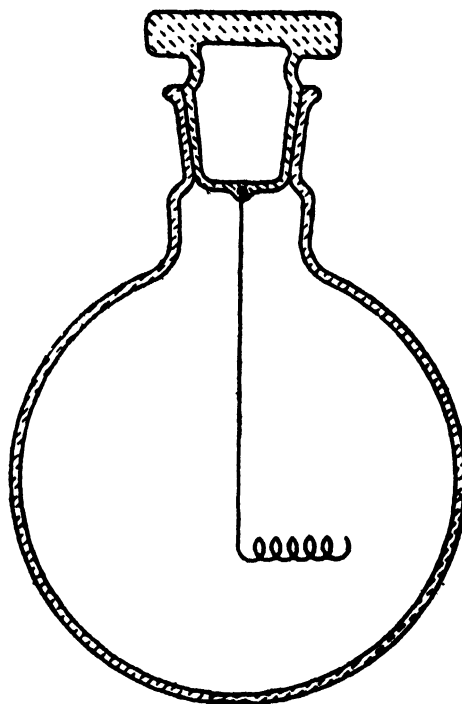


FIG. 2 FLASK FOR OXYGEN COMBUSTION WITH PLATINUM WIRE ATTACHED WITH STOPPER

7.2.2.5 Adhesive tape — of cellulose acetate or regenerated cellulose, coated with polyvinyl acetate, of width 30 mm.

7.2.2.6 Beaker — capacity 100 ml.

7.2.3 Test Sample

7.2.3.1 In case of a liquid — Cut out 30×30 mm square of adhesive tape (see Fig. 3A). Stick on it a square of filter paper with a tail (see Fig. 3B). Wind it around a glass rod, starting at the side parallel and closer to the tail, so as to form a small cylinder, keeping the filter paper inside (see Fig. 3C). Close the extremity of the cylinder opposite the tail by flattening and pinching. Weigh the cylinder to the nearest 0.000 1 g. Then deposit the liquid sample of 0.025 to 0.035 g through the upper end of the cylinder by a tapered pipette. Close this extremity by pinching. Weigh again to the nearest 0.000 1 g. Introduce the whole system within the platinum spiral of the flask with the paper tail protruding. Proceed as in 7.2.4.

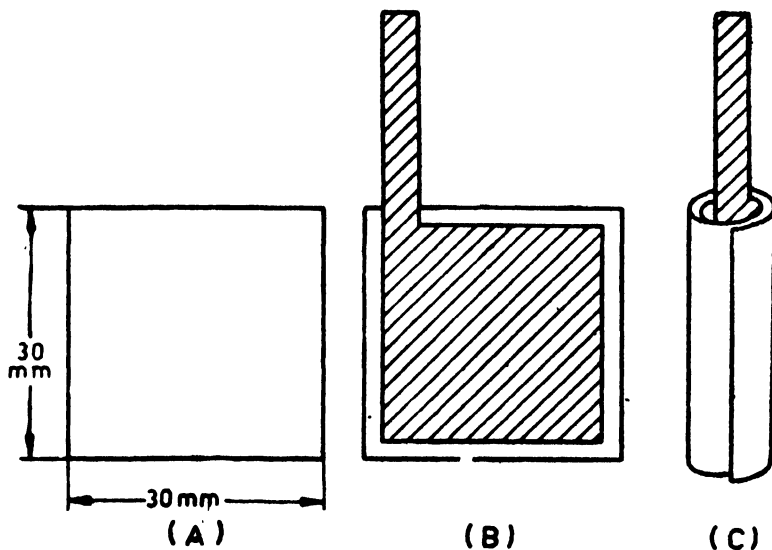


FIG. 3 PREPARATION OF HOLDER FOR LIQUID TEST PORTION

7.2.3.2 In case of a solid — Place 0.025 to 0.035 g of the test sample, weighed to the nearest 0.000 1 g, on a filter paper cut as shown in Fig. 4A and having previously marked folds. Then fold the paper as shown in Fig. 4B, C and D and introduce it within the platinum spiral of the flask, with the paper tail protruding. Proceed as in 7.2.4.

7.2.4 Procedure — Introduce into the flask about 10 ml of water, 1 ml of the potassium hydroxide solution and 0.15 ml of the hydrogen peroxide solution. Pass oxygen into the flask through a glass tube at a rate of 250 to 350 ml/min for 5 min to displace the air. Ignite the filter paper tail with a gas flame and quickly insert the stopper carrying the platinum wire and burning filter paper into the flask. During combustion, keep the flask inverted so that the liquid covers the bottom of the stopper, and leakage of the stopper and escape of gas are avoided. When combustion is finished, turn the flask upright and gently shake under a stream of cold water to cause rapid and complete absorption of the hydrochloric acid produced. After 30 min, open the flask and transfer the contents quantitatively to a 100-ml beaker, rinsing so that the final volume is about 30-ml. Add about 1 g of sodium nitrate and 2.5 ml of the nitric acid solution, and boil the solution for 5 min. After cooling, determine the chlorine content, by electrometric titration or by the Volhard method, with the silver nitrate solution.

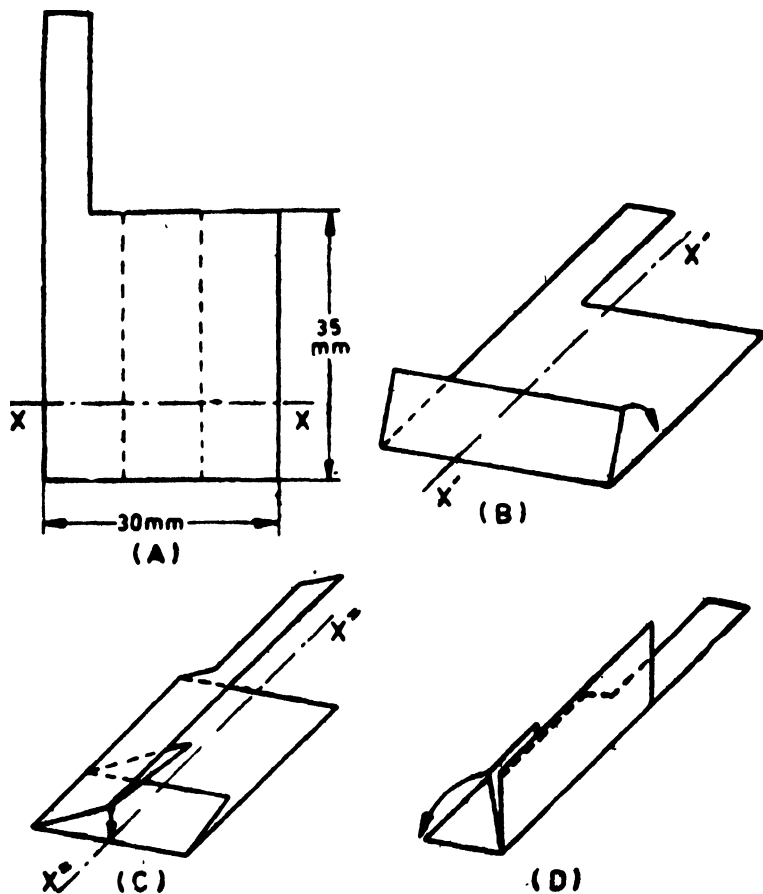


FIG. 4 FOLDING OF FILTER PAPER CONTAINING SOLID TEST PORTION

7.2.4.1 Blank test — Carry out a blank test following the procedure specified in 7.2.3 and 7.2.4 but omitting the test portion.

CAUTION — 1) If a safety cage is not used, it is essential that the operator wears protective goggles and gloves.

2) It is unsafe to hold the flask in the hand during this procedure and it is also dangerous to tool the flask in cold water.

7.3 Expression of Results

7.3.1 The total chlorine content, expressed as a percentage by mass, is given by the formula:

$$3.55 \times \frac{0.1 (V_1 - V_0)}{m}$$

where

V_0 = volume, in ml, of the silver nitrate solution used for the blank test;

V_1 = volume in ml of the silver nitrate solution used for the determination; and

m = mass in grams of the test portion.

7.3.2 Express the result as the arithmetic mean of two determinations which do not differ by more than ± 0.2 percent (m/m).

7.4 Test Report — The test report shall include the following information:

- a) Full identification of the sample;
- b) Test method used (A or B); and
- c) Total chlorine content of the sample, according to **7.3.2**.

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'Pushpak', Nurmohamed Shaikh Marg, Khanpur, AHMEDABAD 380001 550 13 48

‡Peenya Industrial Area, 1st Stage, Bangalore-Tumkur Road,
BANGALORE 560058 839 49 55

Commercial-cum-Office Complex, Opp. Dushera Matdan, E-5 Arera Colony,
Bittan Market, BHOPAL 462016 72 34 52

62/63, Ganga Nagar, Unit VI, BHUBANESWAR 751001 40 36 27

5th Floor, Kovai Towers, 44 Bala Sundaram Road, COIMBATORE 641018 21 88 35

Plot No. 58, Neelam Bata Road, NIT, FARIDABAD 121001 42 82 60

Savitri Complex, 116 G.T. Road, GHAZIABAD 201001 471 19 98

53/5 Ward No.29, R.G. Barua Road, 5th By-lane, Apurba Sinha Path,
GUWAHATI 781003 54 11 37

5-8-56C, L.N. Gupta Marg, Nampally Station Road, HYDERABAD 500001 320 10 84

E-52, Chitranjan Marg, C- Scheme, JAIPUR 302001 37 38 79

117/418 B, Sarvodaya Nagar, KANPUR 208005 21 68 76

Seth Bhawan, 2nd Floor, Behind Leela Cinema, Naval Kishore Road,
LUCKNOW 226001 21 89 23

NIT Building, Second Floor, Gokulpat Market, NAGPUR 440010 52 51 71

Patliputra Industrial Estate, PATNA 800013 26 28 08

First Floor, Plot Nos. 657-660, Market Yard, Gultekdi, PUNE 411037 426 86 59

'Sahajanand House' 3rd Floor, Bhaktinagar Circle, 80 Feet Road,
RAJKOT 360002 37 62 51

T.C. No. 14/1421, University P. O. Palayam, THIRUVANANTHAPURAM 695034 32 21 04

*Sales Office is at 5 Chowringhee Approach, P.O. Princep Street,
CALCUTTA 700072 237 10 85

†Sales Office is at Novelty Chambers, Grant Road, MUMBAI 400007 309 65 28

‡Sales Office is at 'F' Block, Unity Building, Narashimaraja Square,
BANGALORE 560002 222 39 71